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ORIGINAL PAPER

Highly Efficient and Selective Oxidation of Sulfides to Sulfones with Hydrogen Peroxide Under Ultrasonic Irradiation Catalyzed by Copper(II) Schiff Base Complex Supported on Mesoporous MCM-41

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Abstract

In this work, copper(II) Schiff base complex immobilized on functionalized mesoporous silica MCM-41 as a heterogeneous catalyst, MCM-41-pr-NH_2-CuL, was successfully used to oxidize a range of sulfides with 30% hydrogen peroxide under ultrasonic irradiation. The influence of different parameters namely solvent, catalyst amount, and kind of oxidant was investigated in order to find suitable reaction conditions. The obtained results demonstrated that this catalytic system was capable of catalyzing the selective oxidation of different sulfides to sulfones with 30% H_2O_2 under ultrasonic irradiation. The related sulfones were acquired in good to excellent yields (67–98%) in this system. Furthermore, the results indicated that this work could be appropriate for chemo-selective oxidation of sulfides to sulfones in the presence of other functional groups. Simple filtration recovered this catalyst, and no significant loss in its catalytic activity and selectivity was observed after using for five times. Also, no critical alteration was recognized in the FT-IR spectrum of the recovered catalyst after using five times.

Keywords Copper(II) complex · Heterogeneous catalyst · Sulfide · Ultrasonic irradiation · Selective oxidation · Hydrogen peroxide

1 Introduction

Sulfone compounds are important synthetic intermediates owing to their growing usage as agrochemicals, drugs, germicides, and lubricants [1]. The oxidation of sulfides is the main approach for the preparation of sulfoxides and sulfones [2–5]. Nevertheless, the selective oxidation of sulfides to sulfones has been studied to a lesser extent in comparison with the oxidation of sulfides to sulfoxides. It is often noticed that the long reaction times, inappropriate reaction conditions, use of costly oxidants, and low yields of the desired products can be mentioned as disadvantages of sulfide oxidation. Therefore, the main purpose in this area of research has been advancing the perfect catalytic systems for efficient and selective oxidation of sulfides under safe and mild reaction conditions [6]. Schiff base ligands have a crucial role in the development of coordination chemistry due to their simple synthesis, versatility, and easily forming stable complexes with transition metal ions [7–10]. Various applications of transition metal Schiff base complexes in biological and industrial fields such as heterogeneous and homogeneous catalysts, pesticides, antimicrobial and antitumor agents, and solar cells are well documented [11–16].

The principal drawback of homogeneous catalysts is their inconvenience recovery and inconstancy toward oxidative degradation, which limits the effective use of these catalysts in different area such as organic synthesis and chemical industry. The mentioned drawbacks can be solved via immobilization of homogeneous catalysts on solid supports that provides several notable advantages such as simple catalyst separation, long catalytic life, high reusability, and thermal stability [17]. Several methods have been utilized for the immobilization of homogeneous catalysts using different supports such as organic polymers, polystyrene or resins, mesoporous silica (e.g. MCM-41), clay, zeolite Y, etc. [18–23]. In recent years, the focus has shifted toward mesoporous silica MCM-41 as a major support material for the heterogeniztion of homogeneous catalysts. The characteristics of MCM-41 such as great specific surface area (>1000 m^2/g), variable pore diameter (between 1.5 and 10 nm), ease of functionalization, high-thermal stability

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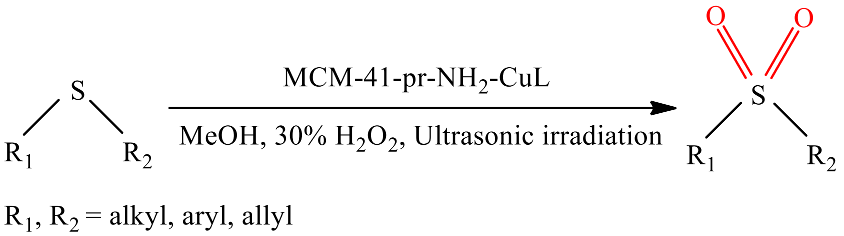
Scheme 1 Oxidation of sulfides with 30% H_2O_2 catalyzed by MCM-41-pr-NH_2-CuL under ultrasonic irradiation



(900 °C), and high-density surface silanol sites make MCM-41 as attractive catalyst support [24, 25].

The essential role of ultrasonic waves in the synthesizing organic compounds is well known. It has been demonstrated that the conversion and selectivity of the wanted product can be improved under ultrasonic irradiation [26, 27]. This enhancement may be due to creating high temperature and pressure local conditions in the reaction mixture and increasing catalyst disaggregation during the sonication process.

Copper is an essential metal in the production of many homogeneous and heterogeneous catalysts particularly for the organic compounds synthesis [28]. In comparison with other transition metals, copper is less toxic, readily available, and cost-effective. The immobilized copper complexes on solid supports such as silica [29], alumina [30], zeolites [31], magnetic nanoparticles [32] and MCM-41 [33–35] have been widely employed as efficient catalysts in different chemical reactions.

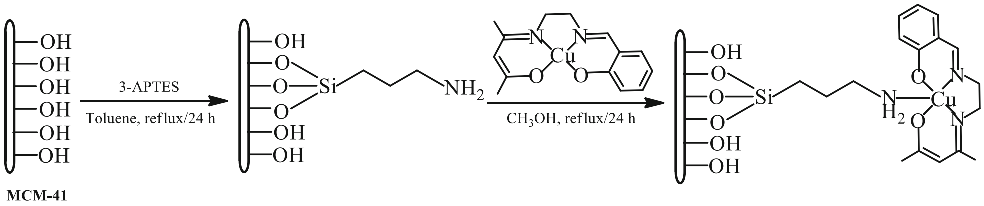
In the present work, we report the selective oxidation of sulfides to sulfones with 30% hydrogen peroxide catalyzed by a copper(II) Schiff base complex immobilized on amino-modified MCM-41 (MCM-41-pr-NH_2-CuL where L = salicylideneiminoethyliminopentane-2-one) under ultrasonic irradiation (Scheme 1). The function of various solvents, oxidant, and amount of catalyst in the catalytic activity of the MCM-41-pr-NH_2-CuL catalyst has been also explored. Moreover, the stability of this heterogeneous catalyst and the ability to reuse it, have been investigated in repeated oxidation reactions under ultrasonic irradiation.

2 Experimental

All substances acquired from Merck and were commercial reagent grade. Sulfides were purchase from Merck or Fluka. Tetradentate Schiff-base ligand, salicylidineiminoethyliminopentane-2-one (H_2L), and its copper(II) complex (CuL) were prepared according to the literature [36]. The MCM-41-pr-NH_2-CuL catalyst was prepared according to our reported procedure [37]. FT-IR spectra were obtained as KBr pellets in the range 400–4000 cm^{-1} with a Thermo SCIENTIFIC model NICOLET iS10 spectrometer. Atomic absorption spectroscopy (AAS) was carried out on a Contra AA300 spectrometer. The catalyst metal content was evaluated by inductively coupled plasma (ICP) on an ICP-Spectrociros CCD instrument. Ultrasonication was performed in a FS 600 ultrasonic processor equipped with 3 mm wide and 150 mm long probe that was immersed directly into the reaction mixture. The operating frequency was 20 kHz and the output power was 0–650 W through manual adjustment. Thin layer chromatography (TLC) on silica-gel polygram SILG/UV 254 plates was used to observe the progress of the reaction. Gas chromatography (GC) experiments were performed on a Philips GC-PU 4600 apparatus equipped with a flame ionization detector (FID) and a 2 m column packed with silicon DC-200.

2.1 General Procedure for the Catalytic Oxidation of Sulfides with H_2O_2 Catalyzed by MCM-41-pr-NH_2-CuL under Ultrasonic Irradiation

In a test tube, sulfide (1 mmol), catalyst (100 mg, 0.012 mmol), and 30% H_2O_2 (2 mmol) were mixed in methanol (2 ml). Then, the reaction mixture was exposed to ultrasonic irradiation for desired time. Checking of the reaction progress was accomplished by TLC using *n*-hexane:ethyl acetate (various ratios). The solid catalyst was filtered after the reaction was completed, and the product was extracted with ethyl acetate (3 × 10 ml) and refined on a silica-gel plate. Finally, the conversion and selectivity values of products were determined by GC.



Scheme 2 The preparation route for the MCM-41-pr-NH_2-CuL catalyst

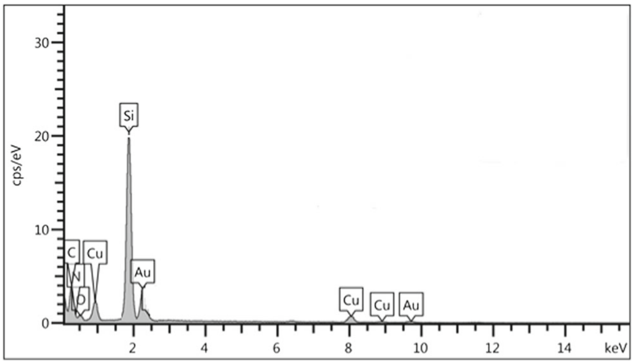


Fig. 1 EDX spectrum of MCM-41-pr-NH_2-CuL

2.2 Catalyst Recycling

To investigate the catalyst recycling, after completion of each reaction, the catalyst was separated from the reaction mixture by simple filtration and carefully washed with methanol. Thereafter, the recycled catalyst was dried in vacuum at 80 °C for 5 h and reused using the same experimental conditions which were described above.

3 Results and Discussion

3.1 Catalytic Oxidation of Sulfides

Scheme 2 demonstrates the catalyst preparation route. The amount of Cu in the catalyst was determined by ICP technique (0.12 mmol per gram of supported catalyst). The energy-dispersive X-ray (EDX) spectrum of the catalyst (Fig. 1) shows the presence of C, N, O, Si and Cu species in the catalyst.

The high catalytic activity of MCM-41-pr-NH_2-CuL in the oxidation of alcohols [37] promoted us to study the ability of this catalyst in the oxidation of sulfides. Also, the reaction yield and selectivity to the desired product can be improved using ultrasonic waves. Therefore, we decided to study the oxidation of different sulfides with H_2O_2 in the presence of MCM-41-pr-NH_2-CuL under ultrasonic irradiation. We selected oxidation of diphenyl sulfide as a model compound, and the impact of reaction parameters including solvent, catalyst amount, and kind of oxidant was investigated to realize the optimal reaction conditions.

Firstly, to select the reaction media, various organic solvents namely acetonitrile, acetone, methanol, ethanol, water, chloroform, and dichloromethane were utilized in the oxidation of diphenyl sulfide with 30% H_2O_2 in the presence of the catalyst under ultrasonic irradiation. The results are shown in Table 1. The data in Table 1 clearly shows that the methanol is a highly effective solvent, producing the product with high selectivity (100%). Hence, we used methanol as solvent for all oxidation reaction of sulfides. The higher conversion and selectivity in the methanol solution is probably due to the better swelling of the catalyst and high solubility of substrates in the methanol media.

Next, the reaction progress was checked with various catalyst amounts. The results (Table 2) showed that the oxidation of diphenyl sulfide did not take place without using a catalyst, while the yield of the reaction increased by increasing the catalyst amount. Based on the obtained results from Table 2, 100 mg (0.012 mmol) of the catalyst is adequate for reaction to be completed.

Finally, the impact of various oxygen-donors such as 30% H_2O_2, urea hydrogen peroxide (UHP), *tert*-BuOOH (TBHP), and $NaIO_4$ on the catalytic activity of MCM-41-pr-NH_2-CuL was studied in the model reaction. The results

Table 1 The effect of solvent on the oxidation of diphenyl sulfide with H_2O_2 catalyzed by MCM-41-pr-NH_2-CuL

Entry	Solvent	Time	(min)	Conversion	(%)^a	Sulfone selectivity	(%)^b
		With US	Without US^c	With US	Without US^c	With US	Without US^c
1	CH_3CN	5	120	73	67	98	80
2	CH_3COCH_3	5	120	42	38	96	75
3	CH_3OH	5	120	98	85	100	83
4	C_2H_5OH	5	120	57	52	100	78
5	H_2O	5	120	52	55	94	77
6	$CHCl_3$	5	120	31	27	87	75
7	CH_2Cl_2	5	120	19	23	100	62

Reaction conditions under ultrasonic irradiation (US): diphenyl sulfide (1 mmol), 30% H_2O_2 (2 mmol), catalyst, MeOH (2 mL)

Reaction conditions without ultrasonic irradiation: diphenyl sulfide (1 mmol), 30% H_2O_2 (2 mmol), catalyst, MeOH (2 ml), $T = 40$ °C

[a,b] Conversion and selectivity determined by GC

[c] Reaction was carried out under conventional mechanical stirring at 40 °C

Table 2 The effect of catalyst amount on the oxidation of diphenyl sulfide with H_2O_2 catalyzed by MCM-41-pr-NH_2-CuL

Entry	Catalyst amount	Time	(min)	Conversion	(%)^a	Sulfone selectivity	(%)^b
	(mg, mmol)	With US	Without US^c	With US	Without US^c	With US	Without US^c
1	0	5	120	7	0	85	0
2	25 (0.003 mmol)	5	120	21	17	96	65
3	50 (0.006 mmol)	5	120	38	29	98	73
4	75 (0.009 mmol)	5	120	53	48	100	77
5	100 (0.012 mmol)	5	120	98	85	100	83
6	125 (0.015 mmol)	5	120	99	83	100	85

Reaction conditions under ultrasonic irradiation (US): diphenyl sulfide (1 mmol), 30% H_2O_2 (2 mmol), catalyst (100 mg), solvent (2 ml)

Reaction conditions without ultrasonic irradiation: diphenyl sulfide (1 mmol), 30% H_2O_2 (2 mmol), catalyst (100 mg), solvent (2 ml), $T = 40$ °C

^{a,b}Conversion and selectivity determined by GC

^{c}Reaction was carried out under conventional mechanical stirring at 40 °C

illustrated that in this catalytic system, the 30% H_2O_2 is ideal oxidant because a higher yield of sulfone product (98%) was obtained (Table 3, entry 1). Furthermore, H_2O_2 is a green oxidant, presenting the superiorities of safety in storage and operation, easy to reach and environmentally safe with the water generation as the only by-product.

With the optimal reaction conditions in hand, oxidation of different kinds of sulfides has been carried out under ultrasonic irradiation. As shown in Table 4, in this catalytic system, different sulfides such as dialkyl and alkylaryl sulfides and sulfides with the various functional groups were successfully oxidized to their according sulfones in good to excellent yields (67–98%) in short reaction times (5–7 min). This oxidizing system allowed the 2-hydroxyl ethyl phenyl sulfide, which has a hydroxyl group, to be chemo-selective oxidized to the corresponding sulfone (Table 4, entry 8). In this case, the alcohol oxidation did not take place under the mentioned conditions and the major product was the sulfone compound which obtained in good yield. The chemo-selectivity of the described system was also studied for the oxidation of sulfides containing C=C bonds (Table 4, entries 10 and 11). In these substrates, the sulfur atom was selectively oxidized and C=C bonds oxidation did not occur under the mentioned reaction conditions.

The main effects of ultrasonic irradiation in the liquid such as a local enhancement in temperature and a local high pressure are due to the cavitation phenomena [38]. These effects led to particle size decrease as well as area enhancement and accelerated the motion of suspended particles leading to a better transfer of liquid-solid mass. Therefore, the use of ultrasonic waves in liquids can be accelerated the reaction speed.

3.2 Comparison of Catalytic Activity of Homogeneous Copper(II) Schiff Base Complex (CuL) and Heterogeneous MCM-41-pr-NH_2-CuL

In order to comparison of catalytic activity of homogeneous copper(II) Schiff base complex (CuL) with heterogeneous MCM-41-pr-NH_2-CuL in the oxidation of sulfides with

Table 3 The effect of various oxidants on the oxidation of diphenyl sulfide catalyzed by MCM-41-pr-NH_2-CuL

Entry	Oxidant	Time	(min)	Conversion	(%)^a	Sulfone selectivity	(%)^b
		With US	Without US^c	With US	Without US^c	With US	Without US^c
1	30% H_2O_2	5	120	98	85	100	83
2	H_2O_2/urea (UHP)	5	120	37	23	95	76
3	*tert*-BuOOH (TBHP)	5	120	54	48	98	73
4	$NaIO_4$	5	120	23	27	82	68
5	No oxidant	5	120	0	0	0	0

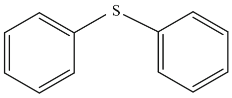
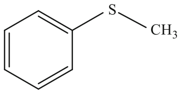
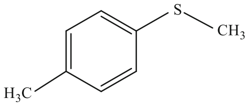
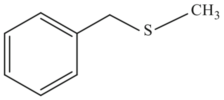
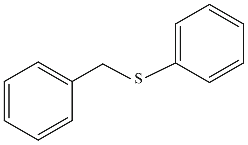
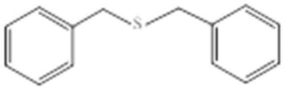
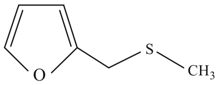
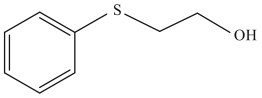
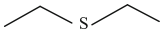
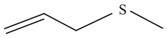
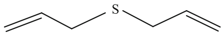
Reaction conditions under ultrasonic irradiation (US): diphenyl sulfide (1mmol), oxidant (2 mmol), catalyst (100 mg), MeOH (2 ml)

Reaction conditions without ultrasonic irradiation: diphenyl sulfide (1 mmol), oxidant (2 mmol), catalyst (100 mg), MeOH (2 ml), $T = 40$ °C

$^{a,\,b}$Conversion and selectivity determined by GC

^{c}Reaction was carried out under conventional mechanical stirring at 40 °C

Table 4 Oxidation of various sulfides with H_2O_2 catalyzed by homogeneous copper(II) Schiff base complex (CuL) and heterogeneous MCM-41-pr-NH_2-CuL catalyst under ultrasonic irradiation

Entry	Sulfide	Time	Conversion (%)[a]		Sulfone selectivity (%)[b]		TON	
		(min)	Heterogeneous	Homogeneous	Heterogeneous	Homogeneous	Heterogeneous	Homogeneous
1		5	98	17	100	98	81.7	13.9
2		5	95	15	100	100	79.2	12.5
3		5	98	23	98	92	80.0	17.6
4		5	87	11	100	95	72.5	8.7
5		5	92	18	94	95	72.1	14.1
6		5	87	27	95	97	68.9	21.8
7		5	74	19	92	93	56.7	14.7
8		5	81	15	93	87	62.8	10.9
9		7	83	13	95	95	65.7	10.3
10		7	72	9	93	85	55.8	6.4
11		7	78	11	89	90	57.9	8.2

Reaction conditions: sulfide (1 mmol), 30% H_2O_2 (2 mmol), catalyst (100 mg of heterogeneous MCM-41-pr-NH_2-CuL or 3.7 mg (0.012 mmol) of homogeneous copper(II) Schiff base complex), MeOH (2 ml)

[a,b]Conversion and selectivity determined by GC

H_2O_2, we repeated all reactions with the same reaction conditions, and the same amounts of catalyst, substrate and H_2O_2. The obtained results are summarized in Table 4. The obtained results showed that in the selective oxidation of sulfides to the sulfones, the turnover numbers (TON) for heterogeneous MCM-41-pr-NH_2-CuL catalyst were higher than the homogeneous copper(II) Schiff base catalyst. One explanation is that attachment of copper(II) Schiff base complex to MCM-41, prevents the formation of μ-oxo catalytically inactive dimers. On the other hand, anchoring the homogeneous catalysts to inert solid supports, increasing the catalytc activity due to the active site isolation which leads to the possibility of the formation of undesired μ-oxo species is minimized [39, 40]. Moreover, the stability of the heterogeneous catalyst is better than the homogeneous catalyst. The heterogeneous catalyst MCM-41-pr-NH_2-CuL can be recovered several times without loss of its activity.

3.3 Catalyst Reusability and Stability

The ability for reusing of a heterogeneous catalyst is one of the most important aspects in economic and environmental terms. To explore the stability and reusability of this catalyst, the reusability of catalyst was examined in the oxidation of diphenyl sulfide with 30% H_2O_2 under ultrasonic irradiation. After the reaction completion, the catalyst was separated by simple filtration, washed

Table 5 Reusability and stability of MCM-41-pr-NH_2-CuL catalyst in the oxidation of diphenyl sulfide with H_2O_2

Run	Time (min)		Conversion (%)[a]		Sulfone selectivity (%)[b]		Cu leached (%)[c]	
	With US	Without US[d]	With US	Without US[d]	With US	Without US[d]	With US	Without US[d]
1	5	120	98	85	100	83	2.7	2.1
2	5	120	98	85	100	80	1.1	0.7
3	5	120	95	83	100	78	0	0
4	5	120	95	78	98	78	0	0
5	5	120	95	78	97	75	0	0

Reaction conditions under ultrasonic irradiation (US): diphenyl sulfide (1mmol), 30% H_2O_2 (2 mmol), catalyst (100 mg), MeOH (2 ml)

Reaction conditions without ultrasonic irradiation: diphenyl sulfide (1 mmol), 30% H_2O_2 (2 mmol), catalyst (100 mg), MeOH (2 ml), $T = 40$ °C

[a,b] Conversion and selectivity determined by GC

[c] Determined by atomic absorption spectroscopy (AAS)

[d] Reaction was carried out under conventional mechanical stirring at 40 °C

thoroughly with CH_3OH, dried carefully, and then reused in the next run. As shown in Table 5, the catalyst could be reused for five times without any meaningful reduction in activity and selectivity. The filtrates after each run were utilized to find the leaching of the catalyst and to determine the Cu leaching using atomic absorption spectroscopy (AAS). The results exhibited that just a little value of the catalyst had been leached in the two first runs (Table 5). Also, no considerable change was observed in the FT-IR spectrum of the recovered catalyst in comparison with fresh catalyst (Fig. 2). In the FT-IR spectrum of recovered catalyst (Fig. 2b), the bands at 473, 801 and 1085 cm^{-1} is correspondence to Si-O-Si vibrations. The two peaks at 1574 and 1605 cm^{-1} are attributed to the two different C=N stretching vibrations of asymmetric Schiff base ligand. Furthermore, the band at 1470 cm^{-1} and two bands at 2941 and 1530 cm^{-1} can be assigned to the aromatic C=C and aliphatic CH_2 groups vibrations, respectively. All of these peaks are presented in the FT-IR spectrum of the fresh catalyst (Fig. 2a). These results imply high stability of the MCM-41-pr-NH_2-CuL catalyst during the oxidation reactions under ultrasonic irradiation.

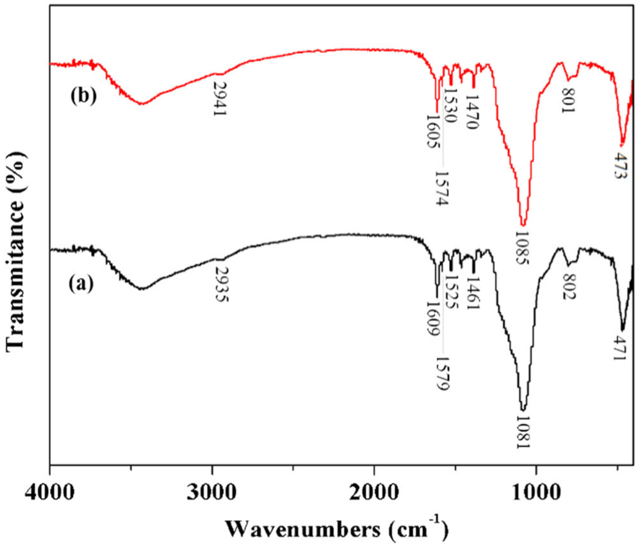


Fig. 2 FT-IR spectrum of **a** fresh MCM-41-pr-NH_2-CuL catalyst and **b** recovered MCM-41-pr-NH_2-CuL

4 Conclusions

As a summary, we have found that copper (II) Schiff base complex which is immobilized on aminopropyl-functionalized MCM-41, MCM-41-pr-NH_2-CuL, can be used as an efficient catalyst for the selective oxidation of sulfides to sulfones using 30% H_2O_2 under ultrasonic irradiation. Good to excellent yields of products, eco-friendly and no by-product formation can be enumerated as the superiorities of this method for the oxidation of sulfides to sulfones. Moreover, the catalyst could be used five times again with no significant decrease in its catalytic functionality and selectivity.

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